# **OXIDATION RESISTANT BRAZING ALLOYS**

GEORGE H. SISTARE, JR.
ALLEN S. McDONALD

HANDY & HARMAN

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#### FOREWORD

This report was prepared by Handy and Harman under USAF Contract No. AF 33(616)-2205. The contract was initiated under Project No. 1252, Task No. 73009, "Brazing Alloy Development", formerly RDO No. 446-48, and was administered under the direction of the Materials Laboratory, Directorate of Research, Wright Air Development Center, with Lt Tsutomu Hikido acting as project engineer.

#### ABSTRACT

During the period from 30 June 1953 to 31 December 1954 some 117 experimental brazing alloys were prepared at the Handy and Harman research laboratories, and evaluated as possible candidates for joining heat conducting metal fins to Inconel tubing for service at temperatures in the order of 1400°F to 1600°F where oxidation resistance of the joint was mandatory.

Alloys of gold-nickel-chromium, and palladium-nickel base alloys with chromium and silicon were developed which can be used to join stainless steel to Inconel at brazing temperatures in the range of 1900°F to 1950°F. The alloys can be torch brazed using flux, or used without flux in a protective atmosphere. The resulting joints resist oxidation at 1600°F service temperature.

It is still possible that with further development palladiumnickel base alloys with silicon-phosphorus-boron additions can be produced which will braze in the temperature range from 1850°F to 1900°F.

The report also contains a note on graphite brazing.

#### PUBLICATION REVIEW

This report has been reviewed and is approved.

FOR THE COMMANDER:

R. W. CONNERS

Lt Colonel, USAF

Asst Chief, Materials Laboratory

Directorate of Research

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This investigation was undertaken for the Wright Air Development Center with the objective of developing brazing alloys for use in joining conductive metal fins to heat resisting metal tubes for service at the highest temperatures at which the fin materials could be used.

The fin materials originally contemplated included: aluminum, silver, copper, sintered aluminum (S.A.P.). oxidation hardened silver-magnesium-nickel—, copper-aluminum, and Inconel clad or stainless steel clad silver or copper. The tube materials involved were Inconel and stainless steel.

Rather early in the program fin materials other than stainless steel clad copper, and oxidation hardened silver-magnesium-nickel were dropped as candidates because they were no longer of interest to the sponsor in connection with this project. Thus the bulk of the investigation was devoted to the development of alloys suitable for joining the latter two materials to Inconel or stainless steel.

Given the general problem of developing a brazing alloy to join two metals for maximum service temperatures, the following factors must be considered. The working temperature 2 must be something less than the melting temperatures of the materials joined, on the one hand, and on the other, should be as high as possible in order that the joints will have sufficient strength at the service temperature. The alloy must be oxidation resistant at the service temperature 2. The alloy must be oxidation resistant at the service temperature 2. The alloy must bond to, and should be fluid on, the metals being joined. For example, a promising silver-aluminum alloy for joining silver-magnesium-nickel to Inconel was deficient in this respect. It would wet and bond at areas of contact with the base metal but was not sufficiently fluid to spread and fill capillary spaces and thus braze in the ordinary sense. The question of fluidity involves the conditions of brazing as to flux and atmosphere. Since it was not desired to use a flux in the ultimate application for which the investigation was undertaken, our efforts were directed toward developing alloys which could be used in a protective atmosphere, although some work

1/Commercial 99.5% silver, 0.3% magnesium, 0.2% nickel hardened by internal oxidation of the magnesium to magnesium oxide.

2/By working temperature we mean the temperature at which a practical braze can be made. What relation the working temperature bears to the liquidus is a function of the constitution of the alloy, the materials joined, the geometry of the assembly, and the conditions under which the joint is made.

3/As an alternative the brazed assembly might be subsequently coated with a protective layer. This possibility was considered in connection with joining hardened silver-magnesium-nickel to Incomel.

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involving fluxes was done. The question of bonding involves the following. The bond must have strength and some ductility. The strength and ductility are not only a function of the properties of the braze metal per se but depend upon the reaction of the braze metal with the base metal. and the properties of the resulting interface. If, for instance, brittle intermetailic compounds are formed at the interface, the joint may be highly unsatisfactory. For example, silver-silicon when used in connection with Incomel forms a brittle interface rendering the joint impractically weak. Fig. 7 is a photomicrograph of such a condition. Aside from the production of such brittle constituents the reaction of braze metal with base metal may result in an unsatisfactory joint due to the extent, and type, of interalloying between braze metal and base metal. The effect (variously referred to as "alloying", "undercutting", or "dilution") is for practical purposes of two general types. In one case it may be a penetrating intergranular attack. On the other hand, it may be general alloying with the surface.

It would be highly desirable if the brazing alloy could be fabricated in wire form or sheet form, but this is not necessary; indeed, most of the commercially available high temperature brazing alloys are not workable, and must be used in powder form.

With these considerations in mind our approach was to investigate certain compositions one might expect to have the proper working temperature and be oxidation resistant or rendered oxidation resistant by a minor addition. The choice of alloy systems and compositions considered was based on our industrial experience in developing brazing materials, and deductions from published constitution diagrams.

The compositions were cast, and if workable fabricated to wire form or sheet form. Specimens from the casting or the fabricated product were exposed to air at elevated temperatures (1300°F to 1600°F depending on the alloy) for times up to seven days. After an oxidation exposure the samples were examined topographically and under the microscope for evidence of oxidation damage. No attempt was made to quantitatively measure oxidation rates. The object of the oxidation exposures was to separate candidates for a practical oxidation resistant brazing alloy from experimental alloys which would be obviously damaged in high temperature service. The criterion of oxidation resistance was the character of the oxidized surface after exposure. If an alloy developed a scale and subscale penetrating into the bulk of the sample as illustrated in Fig. 8, oxidation damage would be expected to continue with time. Alloys developing this type of surface were abandoned as not being oxidation resistant. If, however, after exposure the alloy developed an adherent light surface scale with no evidence of damage below it, as the case illustrated in Fig. 9, the alloy was considered oxidation resistant and given further consideration.

Candidates surviving oxidation tests were used in pad tests (i.e., a test in which a pellet of alloy is melted on a metal pad) involving fin and tube materials as pad, in preparing simple geometry butt and lap joints, and in some cases fin and tube assemblies such as illustrated in Fig. 11. Such assemblies and brazed joints were further exposed to be sure that a joint as well as the braze metal alone was oxidation resistant. In these tests information regarding working temperature, fluidity, and reaction with the base metal was acquired. Promising candidates on the basis of the tests were used to prepare tensile specimens

for short time hot tensile strength determinations. The difficulties of determining the "tensile strength" of a brazed joint, and attaching precise meaning to the observed breaking load of a test specimen prepared for this purpose are well known to those in the industry. The strength of the joint is a function of the brazing alloy, the materials brazed, joint clearance, porosity, etc. The tensile tests made in this investigation are not precision tests and were made only to indicate the order of magnitude of the strength one might expect in practice. All specimens were examined after fracture and only those specimens which in usual industrial practice would be considered to have sound joints were used to furnish data.

Throughout the investigation various aspects of technique occupied our attention, such as the possibility of producing brazing alloy in situ during the brazing cycle, and the possibility of applying a protective coating to the joints subsequent to brazing. The results of such considerations are reported in the sections devoted to the particular alloys in connection with which they were undertaken.



### Silver-Manganese Alloys

### Compositions

Alloy No.	Ag	Mn	<u>Al</u>	Mg
#85 <b>-</b> 15#	85	15	0	0
5176	82	15	3	0
5177	82	15	0	3
5237	80	15	5	0
5238	75	15	10	0

Commercial 85% silver - 15% manganese brazing alloy was an obvious starting point for investigation. For some of the fin materials considered at the inception of the project it would be necessary to both increase the oxidation resistance and reduce the working temperature. connection with the first requirement alloys with 3% of aluminum and magnesium were prepared (#5176, #5177) and exposed to air at 1300°F, together with a specimen of straight silver-manganese ("85-15") for comparison. Inspection of the specimens indicated that the magnesium did not improve oxidation resistance, but that the aluminum modification was markedly better than straight silver-manganese. The magnesium modification was abandoned, and the aluminum increased to 5% and 10% (#5237, #5238) in the aluminum modification. These latter alloys were brittle due to the formation of an aluminum-manganese intermetallic compound. Specimens were exposed to air at 1300°F for 55 hours. Examination of the specimens indicated that although aluminum improved oxidation resistance a sufficient amount could not be incorporated in solid solution due to the formation of an aluminum-manganese compound. The alloys were abandoned because of inadequate oxidation resistance.

### Silver-Aluminum and Silver-Magnesium Alloys

#### Compositions

Alloy No.	Ag	Al	Mg	<u>Zn</u>
5288	95	5	0	0
5289	91	4	0	5
5290	87	3	0	10
5294	92	0	8	0
5295	89	0	6	5
5296	85.5	0	4.5	10
5297	82.5	0	2.5	15

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In this series of alloys the aluminum and magnesium additions were for oxidation resistance, and the zinc was added to increase wettability and reduce the working temperature in connection with the lower melting fin materials contemplated in the early stages of the project. It is a fact based on industrial brazing experience that the presence of zinc in silver brazing alloys increases their ability to wet ferrous metals.

All of the alloys were workable, and were rolled to .032" sheet. Specimens were exposed to air at 1300°F for 55 hours. The silver-magnesium and silver-magnesium-zinc alloys all developed heavy crumbly scales which appeared to progressively penetrate the specimen with time. Fig. 10 is typical of the group. They were abandoned as not being sufficiently oxidation resistant.

The silver-aluminum and silver-aluminum-zinc alloys all form thin, adherent scales. Fig. 9 is typical. No further work was done with the silver-aluminum-zinc alloys because zinc rapidly reduced the melting temperature, and by the time this work was done the lower melting fin materials had been abandoned as possibilities by the sponsor.

The 95% silver - 5% aluminum alloy (#5288) was considered a possible candidate for joining Incomel to oxidation hardened silvermagnesium-nickel. Attempts were made to torch braze lap joints using a flux of lithium fluoride - potassium acid fluoride - potassium chloride. This mixture was adequate to flow the alloy in pad tests. However, when lap joints were attempted large amounts of flux and oxide were included in the joints rendering them very weak as brazed and without any practical strength (i.e., they were very easily fractured by hand) after short oxidation exposures (72 hours at 1400°F). Atmosphere brazing in charcoal purified tank helium was resorted to. Joints were made by preplacing freshly abraded shims of braze metal between plates of hardened silvermagnesium-nickel and inconel and heating to 1600°F in helium. There was extensive alloying of the filler metal with the silver base metal. When attempts were made to braze with filler metal wire, it was found that the metal would not flow, although bonds would form at any points of contact between braze metal and base metal. Presumably the flowing of the braze metal was inhibited by an aluminum oxide skin on the surface. Various attempts to induce flow included the following: plating the braze metal with gold, plating with silver, pickling with sodium hydroxide to remove aluminum from the surface, improving the atmosphere by passing the helium over hot titanium chips and using titanium chips as a getter in the furnace, and using gaseous boron trifluoride flux. None of these attempts were successful. We concluded that it was not feasible to use the alloy in a brazing, situation where capillary flow would be necessary to fill the joint.

I/Sometime later in connection with a similar problem with regard to copperaluminum-tin alloys it was found that sodium fluoride, potassium fluoride and mixtures of the two would flux the surface of aluminum bearing materials. These fluxes were then tried on silver-aluminum and the resulting flow was improved. However, even when so fluxed we found the alloy to have inherently poor fluidity on Incomel.



In the meantime specimens consisting of sandwiches of hardened silver-magnesium-nickel brazed to Incomel using a preplaced .010" shim of braze metal were undergoing oxidation exposures. It was found that on exposure at 1400°F for 190 hours the interface between the braze metal and Incomel remained intact, but the interface between the braze metal and hardened silver-magnesium-nickel developed a fine line of oxidation damage after 24 hours. We concluded that although the braze metal was oxidation resistant and impervious to oxygen diffusion, oxygen diffusing through the silver-magnesium-nickel attacked the silver-aluminum / silver-magnesium-nickel interface.

We considered the possibility of coating hardened silver-magnesiumnickel with aluminum in order to form a silver-aluminum alloy in situ during the brazing cycle, which would both furnish braze metal for the bond, and leave the silver base metal coated with a layer impervious to oxygen diffusion. In this connection .080" sheets of oxidation hardened silvermagnesium-nickel were plated with aluminum for us by the Bureau of Standards (.004" to .005" of aluminum). Using specimens of the plated material in contact with Inconel a bond was achieved in helium at 1550°F. There was considerable alloying of the aluminum with the silver base metal, and compound formation at the Incomel interface. To work out a practical technique for using a method such as this for joining fins to tubing would renuire some meticulous experimentation with regard to the geometry of the assembly, the thickness of aluminum plate, the ratio of plate thickness to total fin thickness, the brazing temperature and time cycle, etc. These details would have to be worked out in connection with a particular assembly. Such work was not undertaken since it was decided that a more attractive alternative approach would be to braze income! to hardened silver-magnesiumnickel with any suitable brazing alloy without regard to oxidation resistance, subsequently coat the assembly with aluminum powder, and diffuse it into the surface.

In this connection -325 mesh atomized aluminum powder and Kestron acrylic resin were obtained from Oak Ridge National Laboratory where these materials had been used for similar purposes. An aluminum paint consisting of powder in the resin vehicle was prepared and painted on specimens of hardened silver-magnesium-nickel butt brazed to Inconel with a brazing alloy consisting of 67% silver, 26.5% copper, and 6.5% palladium. The specimens were diffusion annealed in helium at 1400°F for 2 hours and subsequently heated in air at 1400°F for 40 hours. In this case the alloying of the aluminum with the braze metal was so extensive that in some cases the joints fell apart during the diffusion anneal (presumably due to the formation of a low melting eutectic in the joint). There was also evidence of extensive alloying with the silver base metal. The technique is completely inadequate for application to joints made with silver-copper-palladium. Also the alloying with the silver-magnesium-nickel base metal was sufficiently extensive to indicate that the technique would require fussy control of the aluminum coating even when used with a braze metal not subject to alloying with aluminum.

I/This was one of the experimental alloys (#5284) used in connection with the silver-copper-palladium group discussed below.

### Compositions

Alloy No.	Ag	Au	<u>Al</u>
5313	90	10	0
5314	87.3	9.7	3
5315	85.5	9.5	5
5316	83.7	9.3	7

These alloys were based on the fact that the solution of gold in silver markedly reduces oxygen permeability. Experimental lap joints between 18-8 stainless steel and Incomel were made in helium using 90% silver - 10% gold and exposed to air at 1400°F for 134 hours. The interfaces between braze metal and base metals progressively oxidized with time, and the alloy was abandoned as inadequate.

The three aluminum bearing alloys were abandoned because they were too low melting presumably due to a valley in the liquidus surface of the silver-gold-aluminum system extending from the gold-aluminum eutectic (3.5% of aluminum in gold, melting at 978°F) to the silver-aluminum peritectic (6% of aluminum in silver melting at 1440°F).

### Silver-Palladium and Silver-Palladium-Aluminum Alloys

### Compositions

Alloy No.	Ag	Pd	Al
5317	90	10	0
5318	87.3	9.7	3
5319	85.5	9.5	5
5320	83.7	9.3	7
5398	91.5	2	6.5
5399	88.75	4	7.25

In all cases the alloys of the group were too high melting. The addition of aluminum made matters worse due to the formation of a high melting aluminum-palladium compound. The group was abandoned.



### Compositions

Alloy No.	Ag	Si	Cu <sub>3</sub> P	Ni-P
5666	96.64	3	0.36	0
5667	96.84	3	0	0.16
5668	96.14	3.5	0.36	0
5669	96.34	3.5	0	0.16
5007	98	2	0	0

The silver-silicon alloys were considered as candidates for brazing Incomel to hardened silver-magnesium-nickel in an assembly which would be subsequently coated for oxidation protection. The copper phosphide and nickel phosphide additions were made because silver-silicon is inherently brittle and there is evidence (in the form of research reports in our Company files) that the phosphide additions improve the workability. The alloys proved unsatisfactory because a brittle compound formed at the incomel braze metal interface. Fig. 7 is a photomicrograph of a pad test which was bent slightly. It is clearly seen that the fracture is taking place through the compound at the interface.

Similar brittle interfaces developed in pad tests using the straight 98% silver - 2% silicon alloy.

It was observed that the character of the interface was related to the silicon content. The 3.5% silicon alloys behaved worse than the 3% alloys which were in turn worse than the 2% alloys. In any case, however, the Inconel interfaces were entirely too brittle for practical purposes.

None of the silver-base alloys developed were adequate for brazing hardened silver-magnesium-nickel to Inconel for service in the 1400°F to 1600°F temperature range nor do we see any good prospect for the job. However, if lower service temperatures (in the neighborhood of 1200°F) were of interest some possibilities exist among the standard commercial silver brazing alloys or modifications of them.

### II. SILVER-COPPER-PALLADIUM BASE ALLOYS

### Compositions

Alloy No.	Ag	Cu	Pd	Al	<u>Cr</u>	Si	Mg	Ве
5284	67	26.5	6.5	0	0	0	0	0
5285	60.5	23.5	16	0	0	0	0	0
5286	58.0	26.0	16	o	0	0	0	0
528 <b>7</b>	56.5	27.5	16	0	0	0	0	0
5321	55.9	25.1	16	3	0	0	0	0
5322	54.5	24.5	16	5	0	0	0	0
5323	53.2	23.8	16	7	0	0	0	0
5349	56	26	16	0	2	0	0	0
5350	55	25	16	0	4	0	0	0
5356	56	26	16	0	0	2	0	0
5357	56	26	16	0.	0	0	2	0
5358	56	26	1.6	0	0	0	0	2

This group of alloys is based on the silver-copper eutectic with the addition of palladium to increase the working temperature. The addition of 6.5% of palladium raises the liquidus of silver-copper eutectic from 1435°F to 1500°F, and the addition of 16% of palladium results in an alloy with a liquidus of about 1600°F. The solidus, however, is increased at a lower rate with increasing palladium resulting in alloys with progressively longer melting ranges. This is an undesirable feature of the silver-copper-palladium system.

The straight silver-copper-palladium alloys are inherently not oxidation resistant and developed deep scales and subscales after exposure to air at 1400°F for 55 hours. Fig. 8 is characteristic of the group.

Additions of aluminum, chromium, silicon, magnesium, and beryllium were made for the purpose of improving oxidation resistance. After exposure to air at 1400°F for 16 hours scaling developed. There was evidence that the additions had some effect on the rate of oxidation. For instance, the oxidation front in the 3% aluminum alloy penetrated further than the oxidation front in the 7% aluminum alloy; however, there was no evidence that the character of the oxidation mechanism or the kind of scale produced had been affected, i.e., in all cases the alloy developed heavy, nonadherent

surface scales, and deep subscales which would be expected to progressively penetrate the alloy until the oxidizable constituents had completely burned out. This group of alloys was abandoned as not being sufficiently oxidation-resistant.

III. GOLD PASE ALLOYS

### Compositions

Alloy No.	Au	Ni	Cr
5428	82.5	17.5	0
5429	72	22	6

Both compositions form tough, ductile solid solution alloys which can be worked to wire or sheet. Both alloys are oxidation resistant, and can be used to braze Incomel to stainless steel in helium without flux. Lap joints so made were exposed to air at 1500°F for six days without being damaged. 1 Assemblies similar to those in Fig. 11 consisting of 18-8 stainless steel fins brazed to 3/8" diameter Incomel tubing were prepared in an atmosphere of helium, brazing at 1900°F for 30 minutes. Fig. 12 shows a section from such an assembly brazed with the gold-nickel-chromium alloy after exposure at 1600°F for 88 hours. The assemblies brazed with gold-nickel appear much the same after similar exposures. There is evident some attack of the base metals by the braze metal. How serious a limitation such attack would impose on the application of the alloy would depend upon the geometry and dimensions of the particular assembly under consideration. and the brazing time cycle. On the basis of industrial brazing experience it is the opinion of the authors that the extent of attack is sufficiently slight to consider the alloy feasible for brazing stainless steel and Income! in most applications.

Tensile specimens consisting of butt joints between .060" x 1/2" lengths of 18-8 stainless steel were torch brazed using type "H" Handy Flux. Torch brazing was resorted to, due to space limitations in our experimental atmosphere brazing furnace. However, the quality of the joints as determined by visual inspection was comparable to those made in helium, and there is no reason to suppose that the results of these tests are not valid for brazes made in atmosphere. The specimens were cleaned, excess brazing alloy removed mechanically, and the joint area ground to a slightly reduced section. Specimens were tested at room temperature, 800°F, 1200°F, and 1600°F as brazed and after exposure to air at 1600°F for seven days. The results are given in Figure 1.

I/The point has been raised that the nature of the gold-nickel system is such that alloys based on the system might be oxidation resistant at relatively high temperatures and not so at relatively low temperatures. Above about 1650°F gold and nickel are completely miscible in the solid state. At lower temperatures there is a wide solid solubility gap. It is indeed possible that alloys in equilibrium in this range, and thus consisting of gold-rich and nickel-rich solid solutions, may have different oxidation characteristics. We exposed samples of gold-nickel-chromium to air at 1200°F, 1h00°F, and 1600°F for 17 hours and could see no difference in the extent of surface oxidation under the microscope. However, reactions in the gold-nickel system are sluggish and it is possible that equilibrium was not attained in such short periods of time.

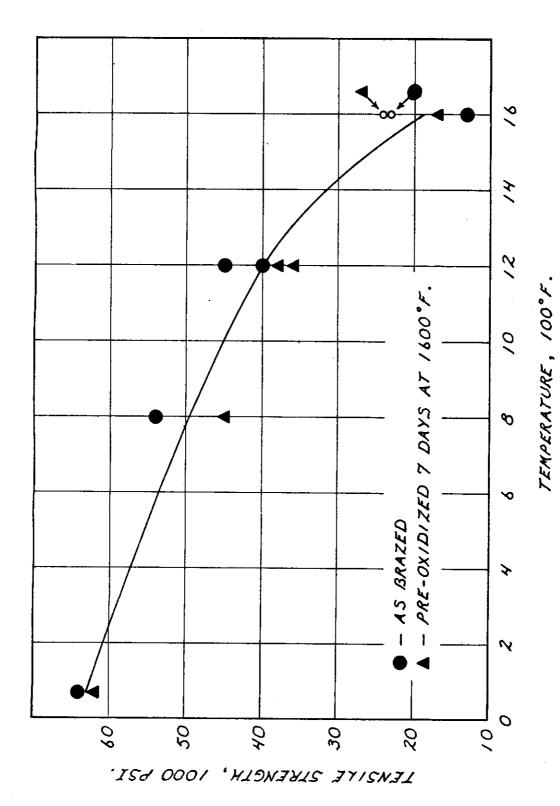


FIGURE 1: HOT TENSILE DATA ON AU-NI-C" ALLOY NO. 5429

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## IV. COPPER-NICKEL-MANGANESE BASE ALLOYS

# Compositions

Alloy No.	Cu	<u>Ni</u>	Mn ·	<u>Cr</u>	<u>A1</u>	<u>Be</u>	<u>si</u>
5300	0	40	60	0	0	0	0
5331	15	31	54	0	0	0	0
5332	25	24.5	50.5	0	0	0	0
5333	35	18.5	46.5	0	0	0	0
5334	45	12.5	42.5	0	0	0	0
5335	55	6	39	0	0	0	0
5336	6 <b>5</b>	O	35	0	0	0	0
							<del></del>
53 <b>37</b>	23	23	49	5	0	Э	0
5338	42.5	11.5	41	5	0	0	0
5346	41	11	<b>3</b> 8	10	0	0	0
<del></del>							
5347	23	23	49	0	5	0	0
5348	42.5	11.5	41	0	5	0	0
					<del></del>	.·. <u>.</u>	
5359	24.5	24	48.5	0	0	3	0
5361	43.8	1.2	41.2	0	0	3	0
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5360	24.5	24	48.5	0	0	0	3
5362	43.8	12	41.2	0	0	0	3
			<del></del>	<u></u>		<u></u>	
536 <b>3</b>	2կ	5]‡	51	0	1	0	0
5364	1414	12	43	0	1	0	0
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5374	0	38.4	57.6	0	4	0	0

The compositions are based on a line extending from the liquidus minimum on the nickel-manganese side of the copper-nickel-manganese ternary (60% manganese - 40% nickel; Liquidus 1864°F) straight across the diagram to the liquidus minimum on the copper-manganese side (65% copper - 35% manganese; Liquidus 1594°F). These basic alloys scaled deeply on exposure to air at 1300°F for 65 hours. Two alloys from the interior of the field (#5332 and #5334) away from the extension of the intermetablic compound on the nickel-manganese side (which made the first two alloys (#5330, #5331) of the basic group very brittle) were chosen for modification. To these selected alloys (#5332, #5334) additions of chromium, aluminum, beryllium, and silicon were made. In all cases after exposure to air at 1400°F for 17 hours there was no evidence of a practical oxidation-resistant alloy resulting. All specimens developed nonadherent scales and subscales.

An additional exploration related to the above was the addition of aluminum to nickel-manganese (#5374). This alloy also scaled after 17 hours' exposure at 1400°F.

These alloys were abandoned as not being sufficiently oxidation resistant.

# V. COPPER-ALUMINUM-TIN ALLOYS

### Compositions

Alloy No.	Cu	Al	Sn
5403	85.5	6.5	8
5404	84.5	6.5	9

The alloys are essentially alpha plus beta aluminum-bronzes which are brittle in the cast state due to the eutectoid decomposition of alpha. However, they can be hot worked. The 9% tin alloy cracked up in processing. The 8% tin alloy was hot worked to .032" gauge. Sandwich brazes were made between stainless steel and Incomel using a preplaced shim of brazing material. As in the case of silver-aluminum alloys the metal does not flow due to the presence of a surface skin of aluminum oxide; however, when preplaced shims are used sound bonding occurs at the interfaces. Unlike the silver-aluminum alloys, however, if a potassium fluoride or sodium fluoride flux is used in an atmosphere of helium or dry hydrogen the metal is extremely fluid on both incomel and stainless steel at a brazing temperature of 1900°F (the silver-aluminum alloys are inherently not fluid on Incomel even when fluxed).

Specimens of lap joints between stainless steel and Incomel were exposed to air at 1500°F for five and six days without oxidation damage at the interface.

Tensile specimens consisting of .060" x 1/2" butt brazes between lengths of 18-8 stainless steel were torch brazed using alloy #5403 and commercial "H" flux. Specimens were tested at room temperature, 800°F, and 1200°F, as brazed and after exposure to oxidation for seven days. The results were very poor. For both as brazed and oxidized specimens the room temperature tensile strength was in the order of 20,000 psi. to 38,000 psi.. the 800°F short time hot strength dropped to the order of 1,500 psi. to 1.800 psi., and the 1200°F strength dropped to the order of less than 1,000 psi. The joints were inspected after testing and found to be very poor as brazes. They were porous, and contained many flux inclusions. To be sure that the unfavorable tensile strengths were not completely the reflection of intrinsically poor joints, butt brazes between plates of stainless steel were made in helium using potassium fluoride flux. Sections cut from these plates were to be used for tensile tests; however, the pieces were very easily fractured at the brazed junction during the manipulation necessary to cut out tensile specimens. Examination of the fractures indicated that the brazes were sound (insofar as they contained few pores).

These alloys were abandoned because of inadequate strength.

### Palladium-Aluminum-Gold

### Compositions

Alloy No.	Pd	Al	Au
5375	92.7	7.3	0
5376	79.5	7	13.5
5377	69.2	6.8	2կ
5378	58.4	6.6	35

This group of alloys is based on the palladium-aluminum eutectic (92.7% palladium - 7.3% aluminum melting at 1873°F) with additions of gold to reduce the working temperature. All of the alloys were flowed on Incomel pads using lithium fluoride-potassium acid fluoride-potassium chloride flux. There was no indication that the gold addition reduced the working temperature. Specimens showed no oxidation damage after exposure to air at 1400°F for 17 hours. None of the group could be flowed on Incomel in helium without a flux. The alloys are inherently brittle and would have to be used in powder form, in which case the presence of an aluminum oxide skin over each particle of powder presents a problem of greater difficulty in the production of practical brazes than the same problem in the case of a workable alloy. In the case of a workable alloy, freshly abraded shims of the rolled braze metal can be preplaced between the members being joined, and flow defects confined to the edges of the assembly, whereas in the case of metal powder, the individual particles must flow and agglomerate if a sound metallic bond is to be achieved. Thus, since we did not think that the alloy could be made to flow in atmosphere without flux, and the use of flux is not wanted in the ultimate application, the group was abandoned.

#### Nickel-Indium and Nickel-Chromium-Indium Alloys

#### Compositions

Alloy No.	Ni	<u>Cr</u>	<u>In</u>	
5424	मिर्ग	46	10	
5427	62	0	38	

The nickel-indium eutectic (#5427) and the nickel-chromium eutectic with an addition of indium to reduce the working temperature were prepared. They were hard, brittle, and unworkable as cast. Samples from the casting were taken for oxidation evaluation. Both alloys showed scale and subscale after 40 hours' exposure at 1500°F due to a nickel-indium compound oxidizing, and were abandoned.

VII. PALIADIUM-NICKEL BASE ALLOYS

# Compositions

Alloy No.	Pd	Ni	Si	<u>Cr</u>	Pd/(Pd + Ni)
Group I.					
5445	58.4	18.1	3.5	20	76
2111146	55.7	17.3	7	20	76
5447	50.4	25.6	14	20	66
5448	47.7	24.3	8	20	66
21111-8	33.1	42.4	4.5	20	fift
5450	31.1	39•9	9	20	मेम
5479	59.5	18.5	7	15	<b>7</b> 6
5480	51	26	8	15	66
5481	33	43	9	15	111
Group II.					
5583	60	40	0	0	60
5584	58	39	3	0	60
558 <b>5</b>	57	38	5	0	60
5586	56	37	7	0	60 <sup>°</sup>
56 <b>05</b>	55	36.75	8.25	0	60
5606	54.5	36	9.5	0	60
Group III.					
5610	26.2	61.1	7.7	5	30
5611	25.9	60.4	8.7	5	30
5612	25.6	59.7	9.7	5	30
5613	25.3	59.0	10.7	5	30
5614	25.0	58.3	11.7	5	30

# PALLADIUM-NICKEL BASE ALLOYS (CONTINUED)

# Compositions

Alloy No.	Pd	Ni	Si	Cr	Pd/(Pd + Ni)
Group III. (Continued)					
5615	24.7	57.6	7.7	10	30
5616	24.4	56.9	8.7	10	30
5617	24.1	56.2	9.7	10	30
5618	23.8	55.5	10.7	10	30
5619	23.5	54.8	11.7	10	30
562 <b>7</b>	39.6	48.4	7	5	45
5628	39.15	47.87	8	5	45
5629	38.7	47.3	9	5	45
56 <b>30</b>	38.25	46.75	10	5	45
5631	37.8	46.2	11	5	45
5632	37.35	45.65	7	10	45
5633	36.9	45.1	8	10	45
5634	36.45	44.55	9	10	45
5635	<b>3</b> 6.0	孙•0	10	10	45
5636	35.55	33.45	11	10	45
5647	53.25	35.50	6.25	5	60
5648	52.65	35.10	7.25	5	60
564 <b>9</b>	52.05	34.70	8.25	5	60
5650	51.45	34.30	9.25	5	60
5651	50.85	33.90	10.25	5	60

### PALLADIUM-NICKEL BASE ALLOYS (CONTINUED)

#### Compositions

Alloy No.	Pd	Ni	Si		Cr	Pd/(Pd + Ni)
	Group III. (Continued)					
5652	50.25	33.50	6	<b>.</b> 25	10	60
<b>5</b> 65 <b>3</b>	49.65	33.10	7	. 25	10	60
5654	49.05	32.70	8	.25	10	60
5655	48.45	32.30	9	.25	10	60
Alloy No.	Pd	Ni_	Si	<u>P</u>	Cr	Pd/(Pd + Ni)
Group IV.						
5693	58.2	38.8	0	3	O	60
5694	57.0	38.0	O	5	0	60
5703	35.1	42.9	8	4	10	45
5704	23.4	54.6	8	4	10	30

All of the compositions are too hard and brittle to work and would have to be used in powder form.

The first six alloys produced (#5445 to #5450) were based on the assumed nature of the palladium-nickel-chromium liquidus surface. The compositions can be rationalized in the following manner. Referring to Figure 2 consider a palladium-nickel-chromium triangle (without regard to silicon content for the moment). All six alloys are on a 20% chromium line. Alloys #5445 and #5446 are in the neighborhood of the intersection of the 20% chromium line with a straight line from the palladium-nickel liquidus minimum at 40% of nickel in palladium on the palladium-nickel side to the point at 38% of chromium in palladium on the palladium-chromium side (where a liquidus minimum in palladium-chromium is reported to exist). Alloys #5447 and #5448 are in the neighborhood of the intersection of the 20% chromium line with a straight line from the point at 40% of nickel in palladium on the palladium-nickel side to the point at 59.5% of chromium in palladium on the palladium-chromium side (where a eutectic is reported to exist). Alloys #5449 and #5450 are in the neighborhood of the intersection of the 20% chromium line with a straight line from the point at 40% of nickel in palladium on the palladium-nickel side to the point at 49% of nickel in chromium on the nickel-chromium side (the nickel-chromium eutectic). To these basic compositions silicon was added to reduce the melting ranges as a result of the formation of the primary nickel-silicon and palladium-silicon eutectics. Roughly two "levels" of silicon were tried; a "lower level" of 3.5%, 4% and 4.5% of silicon and a "higher level" of 7%, 8% and 9% of silicon. These silicon "Levels" are "tilted upward" as one moves away from the palladium corner in accordance with a reasonably assumed upward trend of the liquidus surface as one moves away from the paliadium corner. Thus on the "Lower level" silicon-bearing alloys, #5449 is farther away from the palladium corner than #5447 which in turn is farther away from the palladium corner than #5445. Accordingly, #5449 was made with 4.5% silicon, #5447 with 4% silicon and #5445 with 3.5% silicon. In a similar manner for the "higher level" silicon-bearing alloys, #5450 is farther away from the palladium corner than #5446 which is in turn farther away from the palladium corner than #5446 and accordingly #5450 was made with 9% silicon, #5446 with 8% silicon, and #5446 with 7% silicon.

Pad tests on Incomel in helium atmosphere were made using the above alloys in the temperature range from 1700°F to 1900°F. All of the "lower level" silicon alloys had flow points above 1900°F and were abandoned as being too high melting. The "higher level" silicon alloys began to flow in the region from 1850°F to 1900°F with the temperature at which flow began decreasing in the direction of increasing palladium (i.e., #5446 < #5448 < #5450). At this point alloys #5446, #5448, and #5450 were considered the best candidates. Pad tests with these alloys were exposed to air at 1600°F for 140 hours and found oxidation resistant. An assembly consisting of 1" square 18-8 stainless steel fins brazed to 3/8" diameter Incomel pipe was made(Fig. 11). The relative flow and liquation of the three alloys can be seen by noting the character of the fillets in the picture. Alloy #5446 was chosen as the best candidate. Tensile specimens consisting of butt brazes between .060" x 1/2" lengths of 18-8 stainless steel were made and tested at room temperature. 800°F, 1200°F, and 1600°F as brazed and after exposure to air at 1600°F for seven days. The results are given in Figure 5.

We decided to see if the reduction of chromium content in the "higher level" silicon from 20% to 15% would reduce the working temperature and still result in oxidation resistance. Alloys #5479, #5480, and #5481 were made with this in view. The alloys were as oxidation resistant as #5446 on the basis of exposures to air at 1600°F for 100 hours. We found no practical reduction in working temperature.

We decided to reduce the chromium further, and approach the palladium-nickel-chromium-silicon quaternary from a different point of view, viz.. start from the palladium-nickel-silicon base and add the minimum chromium necessary for oxidation resistance. The approach can be rationalized on the following basis. Referring to Figure 3, there is a primary eutectic in the nickel-silicon system at 11.2% of silicon melting at 2106°F. There is some evidence of a primary eutectic in the palladium-silicon system at 6% silicon melting at 1238°F. One might expect a entectic valley to run from 6% silicon on the palladium-silicon side across the ternary field to 11.2% silicon on the nickel-silicon side. In order to investigate this possibility alloys #5583 to #5586, #5605, and #5606 were cast. The alloys are based on the palladium-nickel liquidus minimum at 40% nickel, and on a line from that point to the silicon corner of the triangle. Indeed, as silicon is added the melting range drops markedly (Alloys #5585 (5% silicon) and #5586 (7% silicon) melt in the neighborhood of 1600°F) and in #5605 with 8.25% silicon a eutectic structure can be seen metallographically. These straight palladium-nickel-silicon alloys are not oxidation resistant. After 67 hours air exposure at 1600°F #5584 was badly scaled, and after 88 hours' air exposure at 1400°F, #5585 and #5586 were badly scaled.

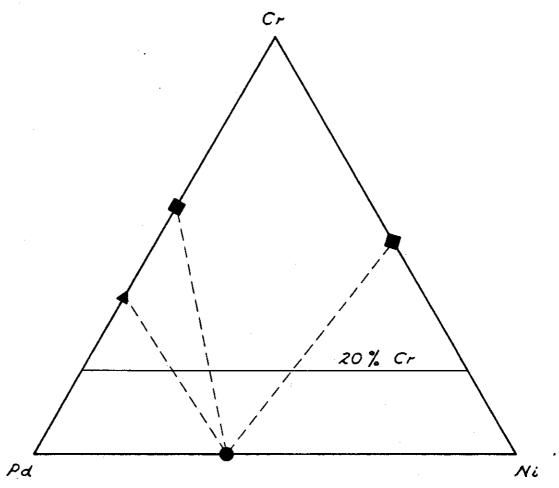


FIGURE 2
PALLADIUM-NICKEL-CHROMIUM DIAGRAM

- Ni-Pa SOL. SOL. MINIMUM: 60% Pa, 2255°F.
- ▲ Pa-Cr SOL. SOL. MINIMUM: 38% Cr, 2372°F.
- Pd2Cr3-Cr EUTECTIC: 59.5 % Cr. 2408°F.
- ♦ Ni-Cr EUTECTIC: 51% Cr, 2449°F.

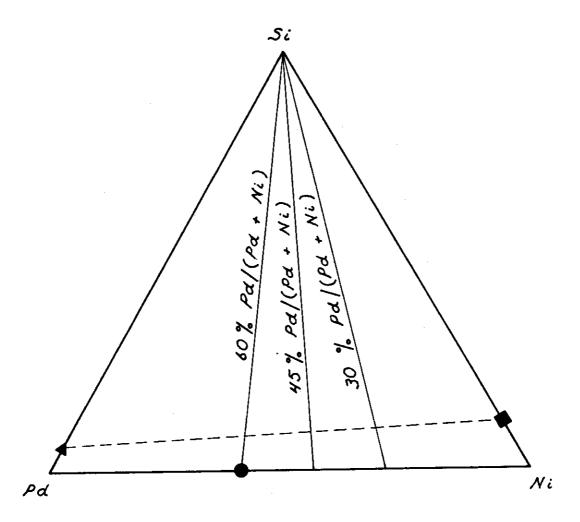


FIGURE 3
PALLADIUM-NICKEL-SILICON DIAGRAM

- Ni-Pd 50L. SOL. MINIMUM: 60 % Pd, 2255°F.
- ▲ Pd-Pd2Si EUTECTIC: 6% Si, 1238°F.
- Ni-Ni3Si EUTECTIC: 11.2 % Si, 2106°F.

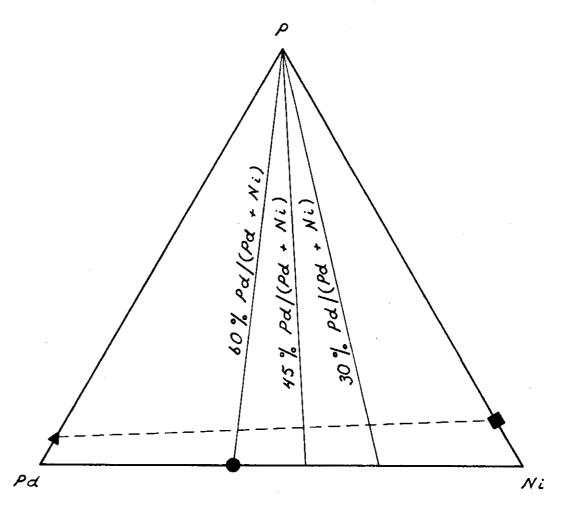


FIGURE 4
PALLADIUM -NICKEL-PHOSPHORUS DIAGRAM

- Ni-Pa 50L. SOL. MINIMUM: 60 % Pa, 2255 °F.
- ▲ PdsP-Pd3P EUTECTIC: 6.4% P, 1450°F.
- Ni-Ni3P EUTECTIC: 11% P, 1616°F.

We next prepared a series of thirty paliadium-nickel-siliconchromium alloys rationalized on the following basis. Referring to Figure 3
we suppose that there is a eutectic valley in the liquidus of the palladiumnickel-silicon ternary extending from 6% silicon on the palladium-silicon
side to 11% silicon on the nickel-silicon side. We suppose that alloys
along this valley will have working temperatures ranging from relatively
low on the palladium-silicon side to relatively high on the nickel-silicon
side. We have found, however, that straight palladium-nickel-silicon alloys
are not sufficiently oxidation resistant, but that an addition of chromium
in the order of 10% will make them so. We have therefore added 5% and 10%
of chromium to our alloys. Since chromium is readily soluble in nickel and
there is evidence in the literature that it has some solubility in palladium,
we further suppose that the 5% and 10% sections of the palladium-nickelchromium-silicon quaternary, except for higher thermal points, will not be
drastically different from the palladium-nickel-silicon face.

Bearing these considerations in mind we have prepared a series of compositions on lines of constant Pd/(Pd + Ni) ratios of 30%, 45% and 60% in the region where these lines intersect a straight line from the 6% silicon on the palladium-silicon side to 11% silicon on the nickel-silicon side.

Pad tests on Incomel sheets illustrated in Figs. 13 and 14 were made in an atmosphere of helium in the temperature range from 1750°F to 2000°F. By studying such specimens certain information as to the nature of the alloy can be determined. For example, in Fig. 13 the relative amount of liquation at any temperature as the silicon content increases from 7% to 9% indicates that the alloys with silicon contents of 8% and 9% are the most eutectiferous in behavior. For any one composition the temperature range between the first sign of melting, and complete melting is an indication of the melting range. For instance, in Fig. 13 the 45% Pd/(Pd + Ni) set shows in general a melting range from 1750°F to 1900°F (some 250°), while in Fig. 14 the 30% Pd/(Pd + Ni) set indicates a much narrower melting range, in the order of 100°, from 1900°F to 2000°F.

These pad tests indicated that the melting range increased with increasing Pd/(Pd + Ni) content, and that working temperature decreased with increasing Pd/(Pd + Ni). The 45% Pd/(Pd + Ni) set was considered a compromise.

With regard to oxidation resistance the examination of specimens exposed to air at 1600°F for 100 hours indicated that the oxidation resistance increased with increasing silicon content, and increasing chromium content; but decreased with increasing Pd/(Pd + Ni) content.

On the basis of these considerations alloys #5633 and #5634 with 45% Pd/(Pd + Ni) content, 10% of chromium, and 8% and 9% of silicon were considered the best candidates.

Tensile specimens consisting of butt brazed .060" x 1/2" lengths of 18-8 stainless steel torch brazed with type "H" Handy Flux were prepared and tested at room temperature, 800°F, 1200°F, and 1600°F, as brazed and after exposure to air at 1600°F for seven days. The results are given in Figure 6. Assemblies consisting of stainless steel clad copper fins brazed to Incomel tubing were made in helium atmosphere in the temperature range



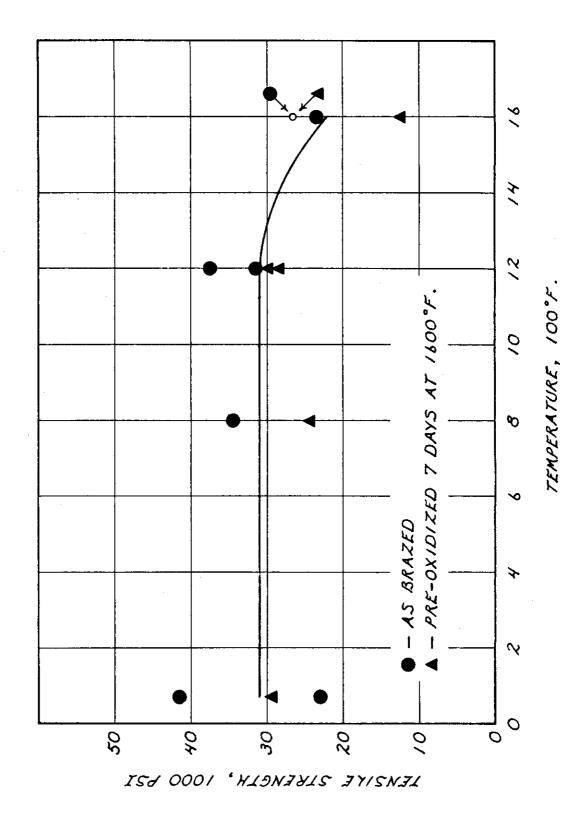


FIGURE S. HOT TENSILE DATA ON PA-NI-Cr-Si ALLOY NO. 5446

WADC TR 55-22

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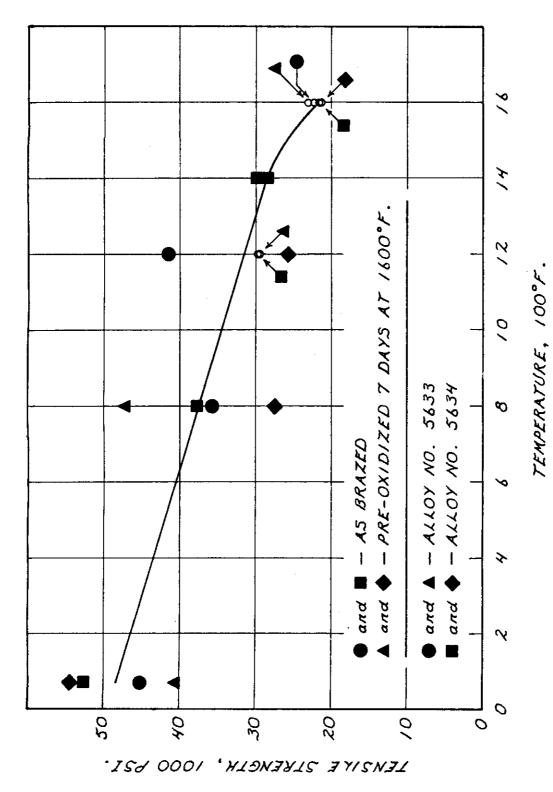


FIGURE 6: HOT TENSILE DATA ON PA-NI-Cr-Si ALLOYS NO. 5633 and 5634

from 1850°F to 1950°F. In the range from 1850°F to 1900°F brazes can be made but a sintered residue remains in the fillet area. In the range from 1900°F to 1950°F good filleting develops. Figs. 15 and 16 illustrate assemblies brazed at 1900°F, 1925°F, and 1950°F.

We chose the 45% Pd/(Pd + Ni) alloys as a compromise between working temperature and melting range, hoping to achieve a low working temperature (in the order of 1850°F to 1900°F) at the expense of a long melting range but since a working temperature of 1900°F is necessary the 30% Pd/(Pd + Ni) group might have been a better choice. The latter group offers a desirably narrow melting range and the working temperature is probably not much over 1900°F.1

Were we to continue on the project we would investigate the 30% Pd/(Pd + Ni) group with additions of phosphorus and/or boron with or without silicon in an attempt to reduce working temperature.

In connection with possible phosphorus additions the exploratory alloys in group IV. were prepared during the terminal stages of the project. These alloys can be rationalized by referring to Figure 4. There is a eutectic on the palladium-phosphorus side of the ternary at 6% of phosphorus. There is a primary eutectic on the nickel-phosphorus side at 11% phosphorus. Our alloys are based on lines of 30%, 45% and 60% Pd/(Pd + Ni) contents in the neighborhood where these lines intersect a line joining these two eutectics.

The addition of phosphorus to the palladium-nickel-chromium-silicon alloys (#5703, #5704) did not reduce the working temperature, but did increase the melting range.

L/Upon formal completion of experimental work on the project and during the preparation of this report a few brazed assemblies were made using the 30% Pd/(Pd + Ni) alloys in the temperature range from 1900°F to 1950°F. Their behavior with respect to flow and filleting was comparable to that of the 45% Pd/(Pd + Ni) alloys in the temperature range from 1850° to 1900°F.

2/Upon formal completion of work on the project and during the preparation of this report the following two alloys were prepared. Alloy #5757; 26.4% palladium, 61.6% nickel, 10% chromium, and 2% boron. Alloy #5758; 23.6% palladium, 55.2% nickel, 10% chromium, 2% boron, and 9.2% silicon. Both alloys contain 30% Pd/(Pd + Ni). Alloy #5757 containing boron without silicon has a working temperature in the neighborhood of 1850°F, is oxidation resistant (no damage after exposure to air at 1600°F for 150 hours), and the preliminary indications are that the melting range is narrow (less than 100°F). Alloy #5758 containing both boron and silicon showed no striking improvement over similar palladium, nickel, chromium, silicon alloys (e.g. #5615,16-17-18-19) without boron.

The addition of phosphorus to the palladium-nickel minimum resulted in alloys with very low solidus temperatures (in the order of 1200°F to 1300°F) and long melting ranges.

The palladium-nicket base alloys are good candidates for brazing Inconel to stainless steel clad copper at brazing temperatures in the range from 1900°F to 1950°F. Good brazes have been made with alloys #5633 and #563h with a 45% Pd/(Pd + Ni) ratio. The alloys with a 30% Pd/(Pd + Ni) ratio have the advantage of a narrower melting range, and their working temperatures are probably not much over 1900°F.

If it is necessary to reduce the working temperature the problem is to do so without too drastically increasing the melting range. Phosphorus was tried in this connection without success. Boron or combinations of boron-phosphorus-silicon still remain possibilities.\*

<sup>\*</sup> After this manuscript was completed a new palladium-chromium diagram was received (E. Raub und W Mahler; Die Palladium-Chrom Legierungen, p. 648, Zeitschrift für Metallkunde, November 1954) indicating that a liquidus minimum does not exist at 38% chromium as indicated in Figure 2, but that the liquidus falls continuously from the melting point of palladium to a eutectic between primary palladium and chromium at about 56% chromium, melting at 2395°F.



### VIII. GRAPHITE BRAZING

During the course of the investigation it was discovered that brazes could be made between graphite and metal, and between graphite and graphite, if during brazing carbide formers are available at the interface. The carbide formers may be furnished by the filler metal or by the base metal. For instance, graphite was brazed to graphite with an alloy of goldnickel with 6% of chromium (#5429) but when the chromium content was reduced to less than 4% bonding did not occur. However, graphite could be brazed to stainless steel with alloys of gold-nickel when the chromium content was reduced to zero, and even with pure gold, presumably because carbide formers were dissolved out of the stainless steel by the filler metal during brazing.

Other alloys found to wet and bond to graphite include commercial 85% silver - 15% manganese, copper-manganese and copper-manganese-zinc containing at least 20% atomic of manganese, and palladium-nickel-chromium base alloys.

Metals we have brazed to graphite include: stainless steel, carbon steel, molybdenum, and copper.

A major problem in graphite brazing is the difference in the thermal expansion between graphite and metal, which produces stresses at the interface during brazing, often resulting in cracks.

Assemblies designed to accommodate the difference in thermal expansion can be adequately brazed.

Fig. 17 illustrates a graphite-to-graphite assembly brazed with gold-nickel-chromium (#5429).

# IX. BIBLIOGRAPHY

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X. APPENDIX - PHOTOGRAPHS

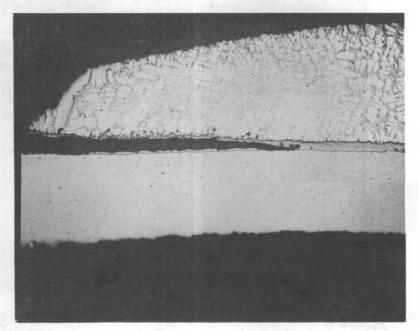


Figure 7

Silver-silicon alloy #5667 flowed on an Incomel pad. The Incomel was subsequently bent slightly initiating the fracture which can be seen proceeding through the brittle compound at the interface.

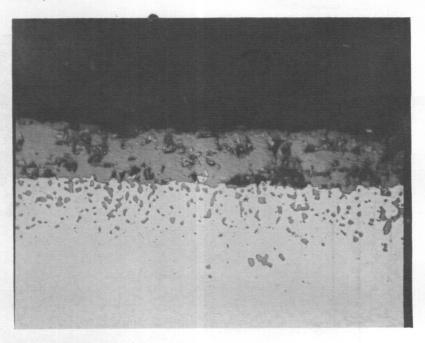


Figure 8 500X

Silver-copper-palladium alloy #5284 after exposure to air at 1300°F for 55 hours illustrating penetrating oxidation damage.

31

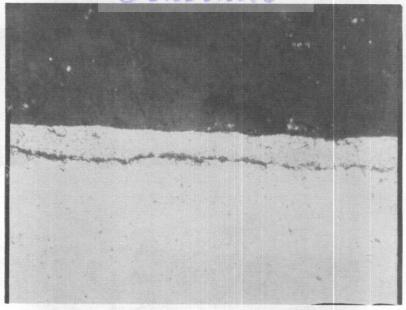


Figure 9

500X

Silver-aluminum alloy #5288 after exposure to air at 1300°F for 55 hours illustrating protective scale.

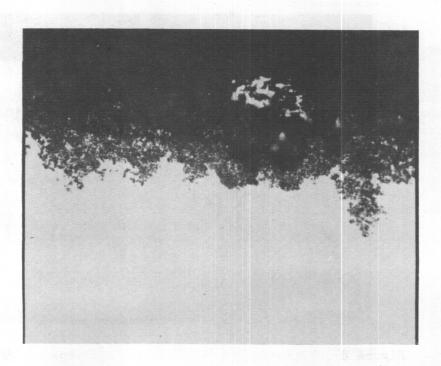
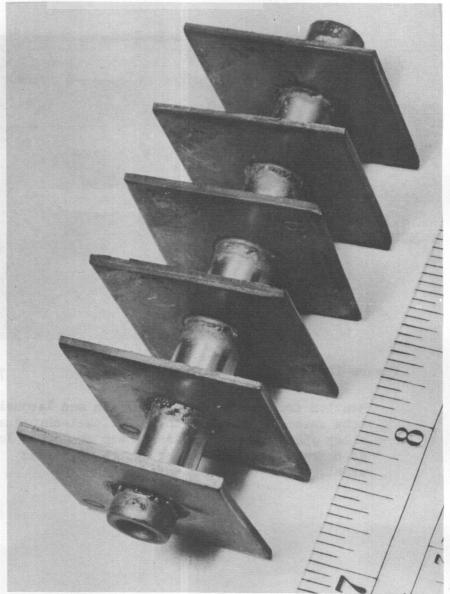


Figure 10

500X

Silver-magnesium-zinc alloy #5229 after exposure to air at 1300°F for 55 hours illustrating oxidation damage.



### Figure 11

Fin and tube assembly consisting of 18-8 stainless steel fins brazed to Inconel tubing. The assembly was brazed with the tube in the vertical position, and the fillet areas in view were on top. The lower two fins (at the bottom of the picture) were brazed with alloy #5450, the middle two with alloy #5446 and the top two with alloy #5448. The relative flow and filleting of the two alloys are illustrated.

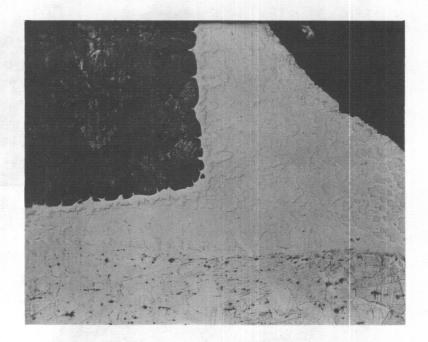


Figure 12

75X

Section from a stainless steel fin and Incomel tube assembly brazed with gold-nickel-chromium alloy #5429 after exposure to air at 1600°F for 88 hours. The dark member is the stainless steel.

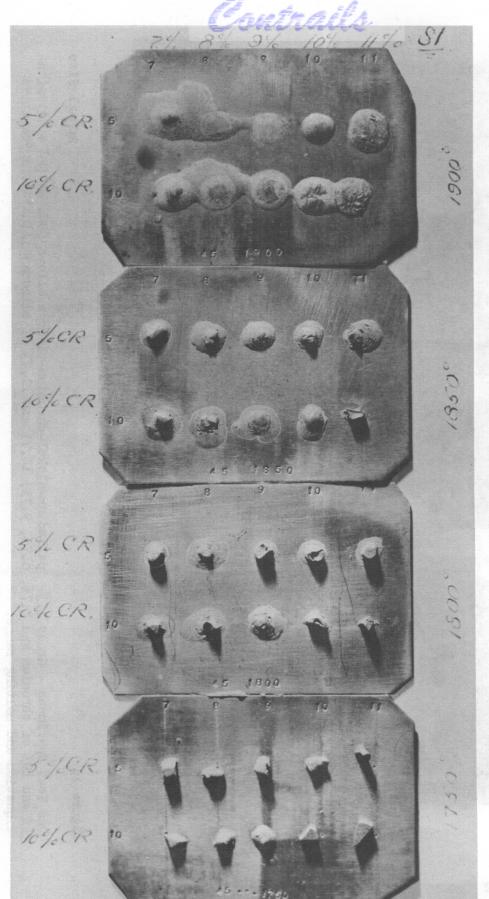


Figure 13

Pad Test Specimens. Group III. palladium-nickel-chromium-silicon alloys (45% Pd/(Pd + Ni) with 5% and 10% of chromium, and 7%, 8%, 9%, 10%, and 11% of silicon) illustrating relative liquation and melting range.

Actual size

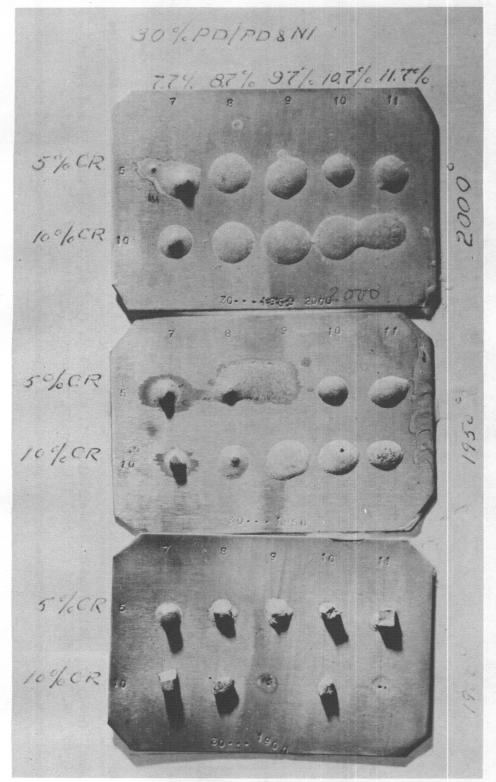


Figure 14

palladium-nickel-chromium-silicon alloys (30% Pd/(Pd + Ni) with 8.7%, 9.7%, 10.7% and 11.7% of silicon) illustrating relative Pad Test Specimens - Group III. 5% and 10% of chromium and 7.7%, liquation and melting range.

Actual Size

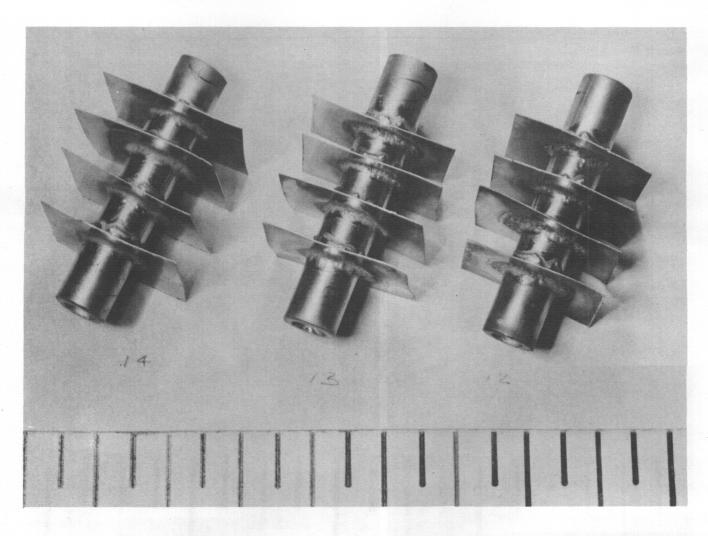


Figure 15 1.5X

Stainless steel clad copper fins brazed to Inconel tubes in helium atmosphere without flux. Top view of fillet area. #12 brazed at 1900°F, #13 at 1925°F and #14 brazed at 1950°F. The upper two fins were brazed with palladium-nickel-chromium-silicon alloy #5703, and the lower two with alloy #5704.

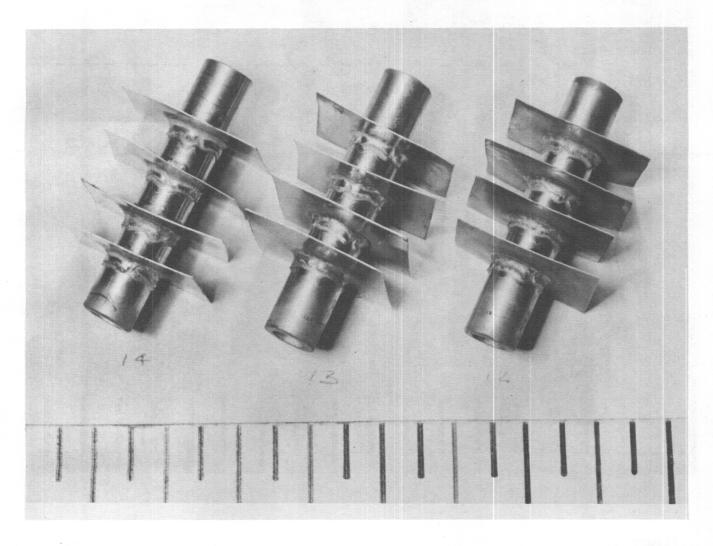


Figure 16

1.5%

Stainless steel clad copper fins brazed to Income tubes in helium atmosphere without flux. Bottom view of fillet area. #12 brazed at 1900°F, #13 at 1925°F, and #14 at 1950°F. The upper two fins were brazed with palladium-nickel-chromium-silicon alloy #5703 and the lower two with alloy #5704.

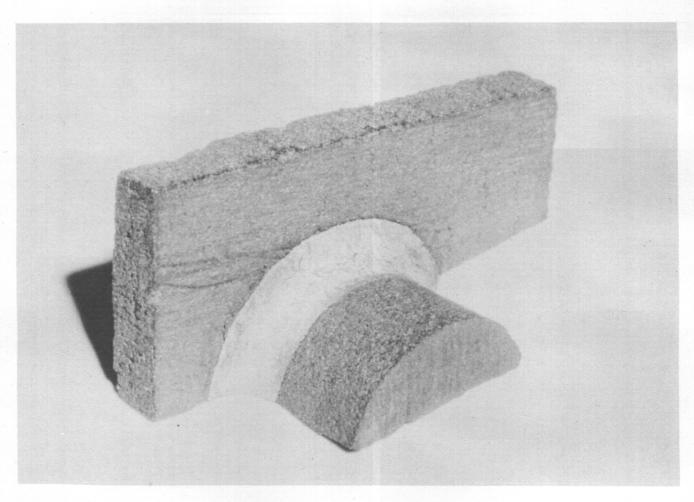


Figure 17

Section from an assembly consisting of a 7/16" diameter graphite rod brazed into a l" square graphite block using gold-nickel-chromium alloy #5429. The braze was made in helium atmosphere at 1900°F to 1925°F.